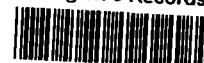




UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, ILLINOIS 60604

EPA Region 5 Records Ctr.



313996

REPLY TO THE ATTENTION OF: SR-6J

VIA ELECTRONIC MAIL AND USPS

Wednesday, June 27, 2007

Linda E Hicken  
RMT, Inc  
744 Heartland Trail  
Madison, Wisconsin 53717-1934

RE: Portage Creek/Kalamazoo River Site  
Plainwell Mill  
Preliminary Comments: Quality Assurance Project Plan

Dear Ms. Hicken:

In September 2006, RMT, Inc. provided a *Draft Quality Assurance Project Plan (QAPP)* on behalf of Weyerhaeuser Company, in accordance with the *Consent Decree For The Design And Implementation Of Certain Response Actions At Operable Unit #4 And The Plainwell Inc. Mill Property Of The Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site (CD)*. Weyerhaeuser and RMT have indicated a change in the format, approach, and scope of the QAPP at the Plainwell Mill. To aid in the expedient revision and quality of revision, the Region 5 is providing preliminary comments and recommendations regarding the draft QAPP, dated September 2006.

1. SOPs from the WATS laboratory have been expired. During the QAPP review time please submit the latest copy of all SOPs which plan to be use for this project.
2. Page 44 of 61. Paragraph 2.10 Data Management Chapter 2.10.2 Laboratory Data Reduction Procedure. Please clarify the definition for the Level 1, 2 and 3 data review.
3. Table 2-2. Summary of Analytical Program for the Groundwater Investigation. The number of samples which will be collected for the all planed analytes (VOCs, PAHs, PCBs, Metals and TSS) are one (1). Do you plan to collect MS/MSD, field blank, field duplicate field equipment blank, trip blank, etc.?
4. Page 6 of 61. U.S. EPA Region 5 QA/QC Coordinator should be changed to U.S. EPA Region 5 QAPP reviewer.

5. Please provide a schedule of work to be performed in graphic or tabular format. The time line must include the start and completion dates for all project activities. (Page 15 of 68 "Instruction on the preparation of Superfund Division Quality Assurance Project Plan," revision 0, June 2000).
6. Which exact Operable Unit will be tested? In the QAPP Units #4, 6 and 7 were found.
7. Submit Chain-of-Custody forms from all laboratories which will participate in the project.
8. Provide the Project/Task Organization Chart during the QAPP review time.
9. The Field/Sampling plan should be more specific. SOPs for field parameters should be submitted.
10. A total of sixteen standard operating procedures for inclusion in the Allied Paper QAPP were reviewed for technical accuracy and agreement with published references. Eleven SOPs are authored by Weyerhaeuser Analytical & Testing Laboratory (WATS); four SOPs are authored by Columbia Analytical Services (CAS) and one SOP is authored by Michigan Department of Environmental Quality (MDEQ). The following comments discuss the findings of these reviews.
- 10.1. General Comments:
  - a. CAS should provide SOP on how they identify and segregate their 'waste' streams.
  - b. The CAS documents submitted have a box for a Document Control Number on the title sheets. The boxes were not completed and no indication was present that the document was an uncontrolled copy.
  - c. Weyerhaeuser's 2 AM E SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP AM E-8082 includes a section titled 'REPORT' which identifies how many significant figures should be used in reporting the data and a section titled 'Key Words'. These sections are not included in SOP AM E-8270. Precision and accuracy discussion is included in Section 10: Quality Control in SOP AM E-8270 but included in Section 11: Report in SOP AM E-8082.
  - d. Weyerhaeuser's 3 AM G SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP AC G-3550 includes sections titled 'Estimate of Preparatory Time' and 'Amount of Sample' not included in the other 2 SOPs. SOP AC G-3550

does not include a section titled 'Key Words' section. SOP AC G-3520 does not include sections titled 'Sample Handling and Storage' or 'References'.

- e. Columbia's 3 SMO SOPs did not include the same number of sections, same section titles nor are the sections in the same order. SOP SMO-GEN does not include a section titled Definitions. SOP SMO-SDIS does not include a section titled Discussions.
- 10.2. Standard Operating Procedure for Sample Disposal (SOP No.: SMO-SDIS, Rev 6, Feb 21, 2006) submitted by Columbia Analytical Services, Inc. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 5.1.2.2 references Attachment I – Potentially Hazardous Materials Criteria, but no copy of the attachment was submitted with the SOP.
- b. Section 5.1.2.3 does not identify the distinguishing characteristics between 'RCRA' waste and 'PCB' waste.
- c. Section 5.2.2 – aqueous samples are held for 31 days but 'solid' samples are held for 2 months. Each month may be either 28 (29 on a leap year), 30 or 31 days long. It would be preferable if the SOP lists a specific number, such as 60 days.
- d. Section 5.2.3.1 indicates that disposal of non-hazardous preserved samples are flushed down the drain with large amounts of water. Preserved samples should be neutralized prior to disposal as preserved samples (acidified metals samples and base preserved cyanide samples are hazardous by pH).
- e. Section 5.2.3.2/5.2.3.3 indicate that solid samples considered non-hazardous are disposed of in the garbage. It is not clear how much testing is done on the samples to determine if any hazardous constituents are present. Since there may be hazardous constituents present which were not tested for, solids should be drummed or lab packed and disposed of as potentially hazardous.
- f. Section 5.2.3.4 does not contain any reference to checks of hazardous constituents other the odor. Foul Condensates should be checked for other hazardous constituents. Also, no check is made to insure that organosulfides are sufficiently neutralized/broken down. The bleach solution pH should then be checked to insure that it is within the correct range for discharge.

- g. Section 5.2.6.2 indicates that containers for non-hazardous samples are dumped in the normal laboratory garbage. Those which contained preserved samples should be triple rinsed and the rinsate neutralized to insure no preservative residue is present in the bottles.
  - h. Section 5.2.6.3 indicates that glass containers which contained hazardous samples are included in the sample waste handling but that plastic containers are dumped in the normal laboratory garbage. Plastic containers should at least be triple rinsed and the rinsate treated as hazardous liquid waste or the plastic bottles treated as hazardous solid waste.
- 10.3. Standard Operating Procedure for Sample Tracking and Internal Chain-of-Custody (SOP No.: SMO-SCOC, Rev 10, March 21, 2006) submitted by Columbia Analytical Services, Inc. The SOP was reviewed for technical accuracy and agreement with published references.
- a. Section 6.5 indicates that, when disposal is performed, each container is scanned. No mention is made regarding how the custody of hazardous samples is handled.

Suggestion:

Include scanning into hazardous waste drum or lab pack.

- 10.4. Standard Operating Procedure for Sample Receiving (SOP No.: SMO-GEN, Rev 22, October 7, 2005) submitted by Columbia Analytical Services, Inc. The SOP was reviewed for technical accuracy and agreement with published references.
- a. Sec 5.1.1 A copy of Table 8-1 from the CAS Quality Assurance Manual should be part of SOP. Does this table indicate a nominal amount of acid or base to be added to the sample in order to preserve the sample properly?
  - b. Section 5.1.8.3 Liquid in coolers from broken samples should be treated as potentially hazardous and disposed of accordingly. Since sample constituents are not known, liquid should never be disposed of down the drain.
  - c. Section 5.1.10/.11 Minimum Chain-of-Custody should include the signing of the Field Chain-of-Custody Record form to acknowledge receipt of the cooler on the COC form. Signing of the COC should never be delayed.

- 10.5. Standard Operating Procedure for Sample Management Procedures (SOP No.: OP SMO-OP, July 15, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

General Comments:

- a. The Weyerhaeuser SOP was issued as a controlled document, with a notation that it was valid for one calendar day from September 16, 2006. The SOP was received by ESAT November XX, 2006, past the valid date of the control number for the SOP. The effective date of the SOP was July 15, 2003. There was no revision number shown on the SOP. There was no indication that the document has been reviewed in the three years since it was issued. The document should be reviewed in accordance with the Weyerhaeuser's quality system, or a frequency recommended by their regulatory agency. A frequency of review/update once every 2 years is recommended in the absence of any other state/ certifying authority requirement. The date of the last revision/review should be noted somewhere in the SOP.

Procedural Review:

- b. Step 3.3.1.f.1 (bullet point 1) states:  
"To take a pH of an aqueous sample, mix the sample by inverting the sample container upside down (to get some sample into the lid). Remove the lid and shake liquid from lid onto front rim of the hood, taking care to avoid direct contact with the lid."

Step 3.3.1.f.2 (bullet point 1) states:  
"...Remove the lid and shake liquid from the lid onto front rim of the hood, taking care to avoid direct contact with the rim.

Step 3.3.1.f.2 (bullet point 2) states:  
"A disposable test tube may also be used, by pouring a very small amount of liquid into the tube. Then dip a pH strip into the tube."

Comment:

The practice of pouring sample directly onto the rim of the hood does not follow the requirements of EPA Method 413.1, is not safe and is likely to result in cross-contamination of samples and possibly spills of hazardous samples. EPA Method 413.1, Step 7.1 states, "...After mixing the sample, check the pH by touching pH-sensitive paper to the cap to ensure that the pH is 2 or lower." Additionally, the Weyerhaeuser SOP does not address clean up of the samples once they have been placed on the rim of the hood.

It should be noted that on April 6, 2004 EPA proposed to withdraw EPA Method 413.1 (69 FR 18165) as an approved method due to its use of an ozone-depleting compound (Freon). When EPA's final rule is approved, Method 413.1 and Standard Method 5520B will be eliminated and only EPA Method 1664A will be approved. If Weyerhaeuser's regulatory permits no longer require the use of EPA Method 413.1, the reference to it and handling samples for analysis by that method should be removed from the SOP.

- c. Step 3.3.1.f.4 states, "Use lead acetate test strips to check for presence of sulfide in liquid samples where cyanide is requested. PbS will precipitate out with color (black). Record pH and sulfide information on Sample Receipt Form)."

Comment:

This statement should be modified to note that if sulfide is present in the sample, it will be indicated by a darkening of the test strip or a dark precipitate on the test strip.

It also should be noted that there is some discrepancy among the reference methods about how lead acetate test strips should be treated prior to use. EPA Methods 335.2 and 335.4 make no mention of any treatment to the test strips prior to use. However, Standard Methods for the Examination of Water and Wastewater, Method 4500, Preliminary Treatment of Samples, states that test strips should be moistened with an acetic acid buffer solution prior to adding a drop of sample. The USEPA Region V Central Regional Laboratory SOP for Cyanide requires that the test strips be moistened with 10% H<sub>2</sub>SO<sub>4</sub> prior to use. Weyerhaeuser may want to consult the manufacturer of the test strips to determine if the test strips require the use of acid to work properly and then incorporate the requirements into the SOP.

- d. Step 3.3.1.f.5 states, "Check other preservation required as specified, such as RCI2 as necessary for N. Carolina regulatory samples."

Comment:

The SOP should clearly define the meaning of the abbreviation RCI2 to ensure that there is no misunderstanding on the part of the reader. It is assumed by the reviewer that RCI2 is referring to residual chlorine. EPA Methods 335.2, 335.4 and Standard Methods 4500 for the analysis of cyanide require checking samples for the presence of oxidizing interferences, such as residual chlorine, by using potassium iodide (KI) test strips. This requirement should be added to the SOP with the same

level of detail as provided for checking for the presence of sulfide. It would be helpful if there was more detail in this section regarding special regulatory requirements.

- e. Step 3.3.1.g states, “ Add any needed preservative in accordance with proper analytical protocol as stated in 40 CFR136.3, Table II.

Comment:

It would be helpful to either add a copy of Table II as an appendix to the SOP or as a controlled document work instruction.

- f. Summary Comments:

In general this SOP seems to be a good, detailed explanation of how samples from several different sources are received and processed into the laboratory. There is sufficient detail to allow personnel to use the SOP as a training document.

The primary issues of concern relate to incorporating sample screening for interferences into the SOP with more detail and resolving the laboratory's practices/requirements for Oil and Grease.

- 10.6. Standard Operating Procedure for Analytical Procedure 3500 (SOP No.: AC G-3500, July 6, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

Series number for some SW-846 Methods have not been updated.

SW-846 5040 should be 5041  
SW-846 8010 should be 8011  
SW-846 8020 should be 8021  
SW-846 8030 should be 8031  
SW-846 8040 should be 8041  
SW-846 8060 should be 8061  
SW-846 8080 should be 8081  
SW-846 8090 should be 8091  
SW-846 8120 should be 8121

- 10.7. Standard Operating Procedure for Sanitation for Soils and Sediments (SOP No.: AC G-3550, December 7, 1993) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 1.3 refers to SOP 4-P-3540 for GPC Clean-up. No copy of this SOP was provided.
- b. Section 7.2 refers to SOP 1-TS-CLP for calculating percent moisture. No copy of this SOP was provided.
- c. Section 7.4 refers to SOP 4-P-CONC for conducting the extract concentration step. No copy of this SOP was provided.
- d. Section 9.3.1 does not identify the volume of surrogates or matrix spiking solution that should be added to the samples.
- e. Section 9.3.4 does not identify what size or type container the extraction solvent should be decanted into.
- f. Section 9.4.3 does not identify the volume of surrogates or matrix spiking solution that should be added to the samples.

- 10.8. Standard Operating Procedure for Extraction by Continuous Liquid-Liquid Extractor (SOP No.: AC G-3520, April 19, 2004) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 6.4 refers to Millipore water that is not listed in section 5.0 Reagents Required.
- b. Section 6 does not identify how extracts will be stored if they are not immediately analyzed.

- 10.9. Standard Operating Procedure for Polychlorinated Biphenyls Analysis by GC/ECD (SOP No.: AM E-8082, June 20, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. No problems observed.



- 10.10. Standard Operating Procedure for Capillary GC/MS for Semivolatile Organics (SOP No.: AM E-8270, July 18, 2003) submitted by Weyerhaeuser Analysis & Testing Laboratory. The SOP was reviewed for technical accuracy and agreement with published references.

Comments:

- a. Section 8.2.1 refers to SOP AC G-ORGSTD for preparing organic standards. No copy of this SOP was provided.
- 10.11. MDEQ SOP #213 (Soil Fraction Preparation for Lead Analysis), Revision 2, Effective Date 01/05 was reviewed for technical accuracy and agreement with published references. There is no published EPA Method for the procedure detailed in this SOP. The SOP references listed in Section 21 were reviewed and used as the standard for comparison. Because the purpose of this SOP is to prepare soil samples collected for MDEQ regulatory programs which monitor exposure to lead, the EPA sample preparation method, EPA Method 3050B, was also consulted for sample requirements.

General Comments:

- a. The MDEQ SOP was received as an uncontrolled copy, but was not marked as such. The SOP document cover page should state whether the copy is controlled or not and should contain a control number or be marked as an uncontrolled copy.

Procedural Review:

- b. Step 14.1 states, "Remove all rocks and vegetation from the soil." This statement needs to be more specific since gravelly soil samples are essentially mostly rocks of various sizes. For example-"Remove all rock and vegetation larger than 2 cm in size from the soil.
- c. Step 14.11 states, "...Place the sieved portion into a mortar and grind with a ceramic pestle; ...." Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must pass through USS Size #10 (2mm diameter particle size). Statement should be revised to say that the sieved portion is ground with a mortar and pestle until all of it passes through a USS Size 10 sieve.
- d. Step 14.11 States, "... (The sieve, mortar, and pestle should be cleaned between samples.)" No specific directions for cleaning these pieces of equipment could be located in the SOP. Must detail method used to clean this equipment (for example-blowing compressed air over it in hood, washing with soap and water, etc.).

- e. Step 14.12 states, "...Place the portion that did not pass through the sieve into a ceramic pestle mortar and grind with a ceramic pestle;..." Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must be ground sufficiently that they will pass through a USS Size #10 sieve (2 mm).
- f. Step 20.1 states, "Generated waste and samples can be thrown out with the trash if they do not contain identified hazardous material (i.e., samples from an industrial site, etc) that require handling as a hazardous waste." All samples should be handled and disposed of as hazardous waste unless proven otherwise.

#### SOP Review

Weyerhaeuser submitted six standard operating procedures for review. In addition, Columbia Analytical submitted four SOPs and Michigan DEQ submitted one SOP for review. No guidance was given for how the reviews were to be completed.

#### Weyerhaeuser

- 11. SOP OP-SMO-OP (Weyerhaeuser Sample Management Procedures SOP) Weyerhaeuser, effective date July 15, 2003, was reviewed for technical accuracy and agreement with published references. There is no published EPA Method for the procedure detailed in this SOP

#### General Comments:

- a. The Weyerhaeuser SOP was issued as a controlled document, with a notation that it was valid for one calendar day from September 16, 2006. The SOP was received by ESAT Dec. 1, 2006, past the valid date of the control number for the SOP. The effective date of the SOP was July 15, 2003. There was no revision number shown on the SOP. There was no indication that the document has been reviewed in the three years since it was issued. The document should be reviewed in accordance with the Weyerhaeuser's quality system, or a frequency recommended by their regulatory agency. A frequency of review/update once every 2 years is recommended in the absence of any other state/ certifying authority requirement. The date of the last revision/review should be noted somewhere in the SOP.

#### Procedural Review:

- b. Step 3.3.1.f.1 (bullet point 1) states, "To take a pH of an aqueous sample, mix the sample by inverting the sample container upside down (to get some sample into the lid). Remove the lid and shake liquid from lid onto front rim of the hood, taking care to avoid direct contact with the lid."

Step 3.3.1.f.2 (bullet point 1) states, "...Remove the lid and shake liquid from the lid onto front rim of the hood, taking care to avoid direct contact with the rim. "

Step 3.3.1.f.2 (bullet point 2) states, "A disposable test tube may also be used, by pouring a very small amount of liquid into the tube. Then dip a pH strip into the tube."

Comment:

The practice of pouring sample directly onto the rim of the hood does not follow the requirements of EPA Method 413.1, is not safe and is likely to result in cross-contamination of samples and possibly spills of hazardous samples. EPA Method 413.1, Step 7.1 states, "...After mixing the sample, check the pH by touching pH-sensitive paper to the cap to ensure that the pH is 2 or lower." Additionally, the Weyerhaeuser SOP does not address clean up of the samples once they have been placed on the rim of the hood.

It should be noted that on April 6, 2004 EPA proposed to withdraw EPA Method 413.1 (69 FR 18165) as an approved method due to its use of an ozone-depleting compound (Freon). When EPA's final rule is approved, Method 413.1 and Standard Method 5520B will be eliminated and only EPA Method 1664A will be approved. If Weyerhaeuser's regulatory permits no longer require the use of EPA Method 413.1, the reference to it and handling samples for analysis by that method should be removed from the SOP.

- c. Step 3.3.1.f.4 states, "Use lead acetate test strips to check for presence of sulfide in liquid samples where cyanide is requested. PbS will precipitate out with color (black). Record pH and sulfide information on Sample Receipt Form."

Comment:

This statement should be modified to note that if sulfide is present in the sample, it will be indicated by a darkening of the test strip or a dark precipitate on the test strip.

It also should be noted that there is some discrepancy among the reference methods about how lead acetate test strips should be treated prior to use. EPA Methods 335.2 and 335.4 make no mention of any treatment to the test strips prior to use. However, Standard Methods for the Examination of Water and Wastewater, Method 4500, Preliminary Treatment of Samples, states that test strips should be moistened with an acetic acid buffer solution prior to adding a drop of sample. The USEPA

Region V Central Regional Laboratory SOP for Cyanide requires that the test strips be moistened with 10% H<sub>2</sub>SO<sub>4</sub> prior to use. Weyerhaeuser may want to consult the manufacturer of the test strips to determine if the test strips require the use of acid to work properly and then incorporate the requirements into the SOP.

- d. Step 3.3.1.f.5 states, "Check other preservation required as specified, such as RCl<sub>2</sub> as necessary for N. Carolina regulatory samples."

Comment:

The SOP should clearly define the meaning of the abbreviation RCl<sub>2</sub> to ensure that there is no misunderstanding on the part of the reader. It is assumed by the reviewer that RCl<sub>2</sub> is referring to residual chlorine. EPA Methods 335.2, 335.4 and Standard Methods 4500 for the analysis of cyanide require checking samples for the presence of oxidizing interferences, such as residual chlorine, by using potassium iodide (KI) test strips. This requirement should be added to the SOP with the same level of detail as provided for checking for the presence of sulfide.

It would be helpful if there was more detail in this section regarding special regulatory requirements.

- e. Step 3.3.1.g states, "Add any needed preservative in accordance with proper analytical protocol as stated in 40 CFR136.3, Table II. "

Comment:

It would be helpful to either add a copy of Table II as an appendix to the SOP or as a controlled document work instruction.

- a. Summary Comments:

In general this SOP seems to be a good, detailed explanation of how samples from several different sources are received and processed into the laboratory. There is sufficient detail to allow personnel to use the SOP as a training document.

The primary issues of concern relate to incorporating sample screening for interferences into the SOP with more detail and resolving the laboratory's practices/requirements for Oil and Grease.

12. SOP AM E-3010 (Acid Digestion of Aqueous Samples and Extracts):

General comment:

- a. The SOP meets the basic technical requirements for SW846 method 3010.

Suggestion:

The SOP should include detailed information about the spiking solutions used. The SOP should include the concentrations used for each elements and how the solution is prepared. This could be accomplished by a solution formula table. The SOP should also state which analytical method the preparation will be used for. (Example: SW846 6010).

13. SOP AM E-3050 (Acid Digestion of Sediments, Soils, and Sludges):  
General comment:

- a. The SOP meets the basic technical requirements for SW846 method 3050.

Suggestion:

The SOP should include detailed information about the spiking solutions used. The SOP should include the concentrations used for each elements and how the solution is prepared. This could be accomplished by a solution formula table. The SOP should also state which analytical method the preparation will be used for. (Example: SW846 6010).

- 14. SOP AM E-160.2 (Suspended Solids in Water and Wastewater): The method requires the sample to be dried to a constant weight. The SOP indicates in Section 8 that a single dried weight is taken. There is no confirmation that constant weight has been achieved. The SOP lists that a control sample is used. There is no information in the SOP as to what the control is or what limits are being used to indicate the batch is in control. All QC limits should be listed in the SOP

15. SOP AI M-ICPMS (Standard Operating Procedure for the Perkin Elmer ELAN 5000 ICPMS):

General comment:

- a. The SOP is written as a general work instruction for operating the PE ICPMS. The SOP references method 200.8 without giving any detail as to how the instrument is used with the method. Digestion SOPs were submitted for the SW846 series, however, the analytical SOP submitted is

for EPA 200.8. The submitted preparation SOPs may be used for methods 6010 or 6020. If method 200.8 is to be used then the preparation procedure for 200.8 must be followed or method 200.2 should be used. Combining of SW846 methods with EPA methods should not be done.

The SOP should give detail on the tuning of the instrument. The method for checking the tune of the instrument is acceptable. All other technical aspects for the work instruction are acceptable. Individual SOPs for method 200.8 and SW846 6020 should be submitted.

16. SOP AQ M-METALS (Metals Team Supplement QZ/QC Guidelines to the A&T):

General Comment:

- a. The SOP details QC criteria for the variety of metals analytical techniques but never states which criteria goes with which methodology. There are no references between the instrument operating instructions and the QC supplement.

Suggestion:

Separate SOPs should be developed based on each test method number. This will reduce confusion as to which method is being followed due to subtle variations in QC requirements.

Columbia Analytical

17. General comment:

The documents submitted have a box for a Control Number. The box was not completed and no indication was present that the document was an uncontrolled copy.

18. SOP SMO-SCOC (Sample Tracking and Internal Chain-of-Custody): Section 6.5 indicates that, when disposal is performed, each container is scanned. No mention is made regarding how the custody of hazardous samples is handled.

Suggestion:

Include scanning into hazardous waste drum or lab pack.

19. SOP SMO-GEN (Sample Receiving): Section 5.1.8.3 Liquid in coolers from broken samples should be treated as potentially hazardous and disposed of

accordingly. Since sample constituents are not known, liquid should never be disposed of down the drain.

- a. Section 5.1.10/11 Minimum Chain-of-Custody should include the signing of the Field Chain-of-Custody Record form to acknowledge receipt of the cooler on the COC form. Signing of the COC should never be delayed.
20. SOP SMO-SDIS (Sample Disposal): Section 5.2.3.1 indicates that the procedure for disposal of non-hazardous preserved samples is to flush the samples down the drain with large amounts of water. Preserved samples should be neutralized prior to disposal as preserved samples (acidified metals samples and base preserved cyanide samples are hazardous by pH).
- a. Section 5.2.3.2/5.2.3.3 indicates that solid samples considered non-hazardous are disposed of in the garbage. It is not clear how much testing is done on the samples to determine if any hazardous constituents are present. Since there may be hazardous constituents present which were not tested for, solids should be drummed or lab packed and disposed of as potentially hazardous.
  - b. Section 5.2.3.4 does not contain any reference to checks of hazardous constituents other than odor. Foul Condensates should be checked for other hazardous constituents. Also, no check is made to insure that organosulfides are sufficiently neutralized/broken down. The bleach solution pH should then be checked to insure that it is within the correct range for discharge.
  - c. Section 5.2.6.2 indicates that containers for non-hazardous samples are dumped in the normal laboratory garbage. Those which contained preserved samples should be triple rinsed and the rinsate neutralized to insure no preservative residue is present in the bottles.
  - d. Section 5.2.6.3 indicates that glass containers which contained hazardous samples are included in the sample waste handling but that plastic containers are dumped in the normal laboratory garbage. Plastic containers should at least be triple rinsed and the rinsate treated as hazardous liquid waste or the plastic bottles treated as hazardous solid waste.
21. SOP MET-1631 (Mercury in Water by Atomic Fluorescence Spectrometry): The SOP meets the technical requirements for EPA method 1631.

#### MDEQ

MDEQ SOP #213, Revision 2, Effective Date 01/05 was reviewed for technical accuracy and agreement with published references. There is no published EPA Method for the procedure detailed in this SOP. The SOP references listed in

Section 21 were reviewed and used as the standard for comparison. Because the purpose of this SOP is to prepare soil samples collected for MDEQ regulatory programs which monitor exposure to lead, the EPA sample preparation method, EPA Method 3050B, was also consulted for sample requirements.

22. General Comments:

The MDEQ SOP was received as an uncontrolled copy, but was not marked as such. The SOP document cover page should state whether the copy is controlled or not and should contain a control number or be marked as an uncontrolled copy.

23. Procedural Review:


- a. Step 14.1 states, "Remove all rocks and vegetation from the soil." This statement needs to be more specific since some soil samples may be mostly rocks of various sizes. For example-"Remove all rock and vegetation larger than 2 cm in size from the soil.
- b. Step 14.11 states, "...Place the sieved portion into a mortar and grind with a ceramic pestle; ...." Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must pass through a USS Size #10 (2mm diameter particle size) sieve. Statement should be revised to say that the sieved portion is ground with a mortar and pestle until all of it passes through a USS Size 10 sieve or equivalent.
- c. Step 14.11 States, "... (The sieve, mortar, and pestle should be cleaned between samples.)" No specific directions for cleaning these pieces of equipment could be located in the SOP. The SOP must detail the method used to clean this equipment (for example-blowing compressed air over it in hood, washing with soap and water, etc.).
- d. Step 14.12 states, "Place the portion that did not pass through the sieve into a ceramic pestle mortar and grind with a ceramic pestle; " Directions are too subjective as written. To meet requirements of EPA Method 3050, samples must be ground sufficiently that they will pass through a USS Size #10 sieves (2 mm).
- e. Step 20.1 states, "Generated waste and samples can be thrown out with the trash if they do not contain identified hazardous material (i.e., samples from an industrial site, etc) that require handling as a hazardous waste." It is not clear how much testing is done on the samples to determine if any hazardous constituents are present. Since there may be hazardous constituents present which were not tested for, solids should be drummed or lab packed and disposed of as potentially hazardous.



Please note that these recommendations do not constitute Region 5's comments to the *Work Plan or Sampling and Analysis Plan*.

Thank you for your attention to this matter. Please do not hesitate to call me at 312.886.1434, should you have any questions related to the task.

Sincerely,



Sam Chummar, Remedial Project Manager  
U.S. EPA Region 5  
Superfund Division – Remedial Response Branch #1  
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